

<p>98-348421/30 E19 J01 FARB 96.12.10 BAYER AG *WO 9825889-A1 96.12.10 96DE-1051216 (98.06.18) C07C 263/20, B01D 9/00, 9/04 Single or multi step suspension crystallisation method - involves final separation of a suspension, at a temperature not more than 8.5 degrees Centigrade lower than the m. pt. of the crystalline phase, in a wash column. (Ger) C98-107722 N(AL AM AT AU AZ BA BB BG BR BY CA CH CN CU CZ DE DK EE ES FI GB GE GH HU ID IL IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MD MG MK MN MW MX NO NZ PL PT RO RU SD SE SG SI SK SL TJ TM TR TT UA UG US UZ VN YU ZW) R(AT BE CH DE DK EA ES FI FR GB GH GR IE IT KE LS LU MC MW NL OA PT SD SE SZ UG ZW) Addnl. Data: DROPE R, GRENNER D, HETZEL H, SCHAL H, WEGENER G 97.11.27 97WO-EP06610</p>	<p>E(10-A14A) J(1-B)</p> <p>separated from the suspension and is re-suspended in a mother liquor at a temperature not more than 8.5°C beneath that of the m. pt. of the crystal phase before introduction into the wash column.</p> <p><u>USE</u> The process is useful for the separation of crystallisable organic compounds from a liquid mixture.</p> <p><u>ADVANTAGE</u> The process enables separation of crystallisable material at a temperature not more than 8.5°C lower than the m. pt. of the crystal phase.</p> <p><u>PREFERRED PROCESS</u> The separated crystal phase is resuspended in the initial isomer mixture. The mother liquor is recirculated between the wash column and the re-suspending vessel and is at a temperature such than the resulting suspension is under saturated. The initial mixture of compounds is a mixture of isocyanate isomers.</p>
<p>A crystallisable organic compound (I) is separated from a liquid mixture of compounds by a single or multi-step suspension crystallisation process. The crystals are separated after the final step in a wash column and a mother liquor that is depleted of (I) by crystallisation is removed in the first step. The crystal phase is</p>	<p>WO 9825889-A+</p>

EXAMPLE

Diphenylmethane diisocyanate (MDI) was produced as an initial isomer mixture of 90% 4,4'-MDI and 10% 2,4'-MDI at a temperature of 36°C. Pure 4,4'-MDI has a m.pt. of 40°C and the isomer mixture depleted of 4,4'-MDI and containing 55 wt.% MDI and the crystal suspension were at 18°C. The separated crystals were fed to a mixing vessel and the initial isomer mixture at 50°C was fed with the mother liquor at a ratio of 100 pts.:70 pts. to a wash column at 34 °C. 77 pts. 4,4'MDI was removed from the head of the wash column per 100 pts. initial isomer mixture. 110 pts. mother liquor was fed from the wash column to the crystalliser and 70 pts. were recycled to the mixing vessel.
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